

**Micro-Pak Enhanced Packaging Stickers:
Enforceable Analytical Method**

DATA REQUIREMENTS:

OPPTS 830.1800

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STUDY COMPLETION DATE:

~~11/29/2012~~ 2/1/13 - KSB

PERFORMING LABORATORY:

Chemir Analytical Services

LABORATORY TEST IDENTIFICATION:

Sodium Metabisulfite by ICP-OES

DATA SUBMITTER:

Micro-Pak Ltd.



STATEMENT OF DATA CONFIDENTIALITY CLAIMS

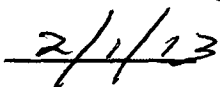
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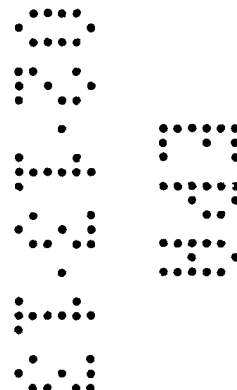
Company: **Micro-Pak Ltd.**

Company Agent: **Robert S. Brennis
Brennis Consulting Services LLC**

Title: **Study Submitter and Agent for
Micro-Pak Ltd.**

Signature: 

Date: 



GOOD LABORATORY PRACTICE COMPLIANCE STATEMENT

This report only concerns the development of an enforceable analytical method for this product. There are no Good Laboratory Practice Standards (GLPS) specifically identified for the development of this data. Therefore, the Good Laboratory Practice Standards (GLPS), as specified in 40 CFR Part 160, are not applicable.

Author: John V. Simpson Date: 2-1-13

Name of Signer: John V. Simpson

Name of Company: Chemir Analytical Services

Company: Micro-Pak Ltd.
Company Agent: Robert S. Brennis
Brennis Consulting Services LLC

Title: Study Submitter and Study Sponsor
Agent for Micro-Pak Ltd.

Signature: Robert S. Brennis

Date: 2/1/13

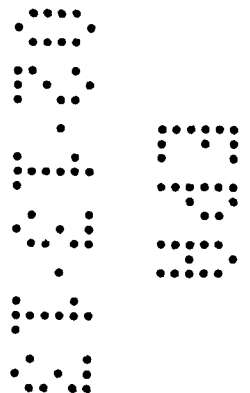
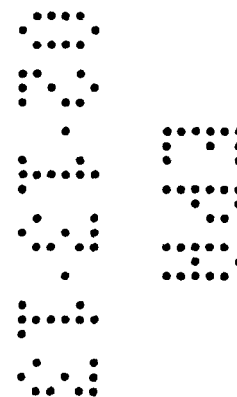


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OPPTS GUIDELINE 830.1800

ANALYTICAL METHOD

An analytical method to verify the certified limits for the total amount of Sodium Metabisulfite in the Enhanced Packaging Stickers is presented below. This method uses a microwave enhanced digestion followed by inductively coupled plasma-optical emission spectroscopy (ICP-OES) to quantify the amount of sulfur present in Micro-Pak Enhanced Packaging Stickers. The sodium metabisulfite concentration is then calculated from this determined sulfur value.

Micro-Pak Ltd.

Quality Control Procedure

Method Introduction

Samples are dissolved using microwave assisted digestion in the presence of acid. Aqueous samples are aspirated and converted to an aerosol through a nebulizer and directed into an argon based plasma. Here the sample is dried, vaporized, atomized and ionized in the argon plasma which generates emission spectra. The resulting light emissions are then directed through a series of lenses where the emitted light is focused onto a detector, measuring the wavelengths characteristic of the elements present. The constituents of an unknown sample can then be identified and quantified. ICP-OES is ideal for determining major and minor concentrations of a wide range of elements in a diversity of sample matrices

List of Chemical Reagents

The following comprises the chemical compounds used during this procedure:

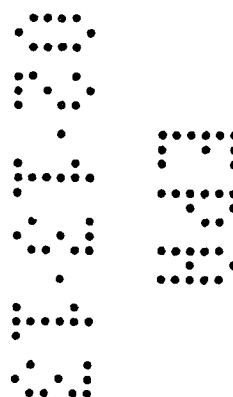
Nitric Acid (68-70%)
Phosphoric Acid (85.6%)
Peroxide (30%)
Deionized water
10031-mg/L Sulfur stock solution (Inorganic Ventures Lot E2-S01120)
1001-mg/L Scandium solution (Inorganic Ventures Lot F2-SC02097)

List of Materials

Volumetric glassware as needed
Microwave digestion tube compatible with Anton Paar Multiwave 300
50ml test tube

Instruments

The following comprises the instruments used during this procedure and all instrumental settings:



Anton Paar Multiwave 300 (SN:80839927) microwave with a HF 100 rotor (SN:80122555)
 Programed to perform the heating curve outlined in table one.

Table 1: Microwave digestion program

| Phase | Temp. (°C) | Ramp (min) | Hold (min) | Fan |
|-------|------------|------------|------------|-----|
| 1 | 200 | 10 | 30 | 1 |
| 2 | 0 | - | 30 | 3 |

Perkin Elmer Optima 7300V ICP-OES set to the conditions outlined in table 2. The detection parameters for each element used in the analysis is shown in table 3.

Table 2: ICP-OES instrumental conditions

| Condition | Setting |
|--------------------|---------------------------------|
| Software | WinLab32 ICP Version 5.1.3.0550 |
| Nebulizer | Teflon GemCone |
| Spray Chamber | Cyclonic |
| RF power | 1300 W |
| Ar Flow | 15.0 L/min |
| Auxillary Ar Flow | 0.2 L/min |
| Nubulizer Gas Flow | 0.80 L/min |

Table 3: Spectroscopic Analysis Parameters

| Element | Wavelength (nm) |
|------------|---------------------------|
| S | 181.975, 180.669, 182.563 |
| Sc (as IS) | 424.683 |

Standard Preparation and Calibration

At each test period, prior to sample analysis, a standard calibration curve is generated using a 20, 100, and & 500 mg/L solution. The standard is prepared by volumetric dilutions from a 10000 mg/L sulfur stock solution (Inorganic Ventures Lot: E2-S01120) along with an addition of a 1000 mg/L scandium internal standard solution to result in a 1mg/L concentration. A quality control standard was also prepared at a concentration of 100 mg/L from a 10000 mg/L sulfur solution procured from a second source (SCP Science Lot: S120116017) as the previous calibration standards.

Sample Preparation

Approximately 0.1 g of the sample is transferred to a microwave digestion tube. A digestion solution was then added to the tube consisting of 5 ml of HNO₃ (68-70%), 2 ml of H₃PO₄ (85.6%), 1 ml H₂O₂ (30%), and 5 ml of deionized water. The mixture is then microwaved according to the method outlined in table 1 using a Anton Paar Multiwave 300 (SN:80839927) with a HF 100 rotor (SN:80122555).

Once digestion is completed, the sample solution is then transferred to a 50 ml test tube and 0.05 ml of a 1000 mg/L scandium internal standard. Then the solution was brought to volume with water.

Each sample was prepared in triplicate and analyzed in triplicate. Due to the nature of the sample matrix, a spike could not be performed. The samples were evaluated against a linear curve having a minimum calibration coefficient of 0.999. Sulfur content was converted to weight percent in the software using the following calculation:

$$\text{Sulfur content (ppm)} \times \frac{\text{Initial sample volume (L)}}{\text{Sample mass (mg)}} \times 100\% = \text{Sulfur content (\%)}$$

A second source QC solution was prepared at a concentration of 99.6 mg/L using a 9960-mg/L S stock solution (SCP Science Lot S120116017). This QC standard was run at the beginning and end with an acceptance criterion of $\pm 10\%$.

Calculation of Weight Percent Active

From the determined sulfur concentration, the amount of sodium metabisulfite present in the same was calculated using the assumption that all sulfur present was in the form of sodium metabisulfite (see calculation below).

$$\text{Metabisulfite Conc. in Sample (mass\%)} = \text{Sulfur Conc. (\%)} \times \frac{\text{Sodium metabisulfite mass (190.107 g/mol)}}{\text{Sulfur mass } \left(32.06 \frac{\text{g}}{\text{mol}}\right) \times 2 \text{ sulfur}}$$

Method Performance

Table 2 lists the wavelengths used during the analysis. The reported values are the result of the average of all wavelengths listed in Table 2 and have a maximum RSD of 3%. The reported values of the analysis of two samples are present in Table 4.

Table 4: Sodium Metabisulfite concentration replicate analysis

| Sample A | | | | | Sample B | | | | | |
|----------|-----|-------------------------|-------|-------|----------|---------|-------------------------|-------|------|--|
| Prep. | Run | Metabisulfite Conc. (%) | Avg. | %RSD | Prep. | Run | Metabisulfite Conc. (%) | Avg. | %RSD | |
| 1 | 1 | 19.57 | 18.29 | 6.19 | 1 | 1 | 20.16 | 19.42 | 3.31 | |
| 1 | 2 | 17.91 | | | 1 | 2 | 19.06 | | | |
| 1 | 3 | 17.40 | | | 1 | 3 | 19.03 | | | |
| 2 | 4 | 18.83 | 18.11 | 6.34 | 2 | 4 | 20.34 | 19.79 | 3.20 | |
| 2 | 5 | 16.78 | | | 2 | 5 | 19.92 | | | |
| 2 | 6 | 18.71 | | | 2 | 6 | 19.09 | | | |
| 3 | 7 | 18.95 | 17.47 | 12.46 | 3 | 7 | 20.04 | 19.83 | 1.48 | |
| 3 | 8 | 14.97 | | | 3 | 8 | - | | | |
| 3 | 9 | 18.50 | | | 3 | 9 | 19.63 | | | |
| Average | | 17.96 | | | | Average | | 19.66 | | |
| %RSD | | 7.823 | | | | %RSD | | 2.716 | | |

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